

# **Probing the Surface of $\gamma$ -Al<sub>2</sub>O<sub>3</sub> by Oxygen-17 Dynamic Nuclear Polarization Enhanced Solid-State NMR Spectroscopy**

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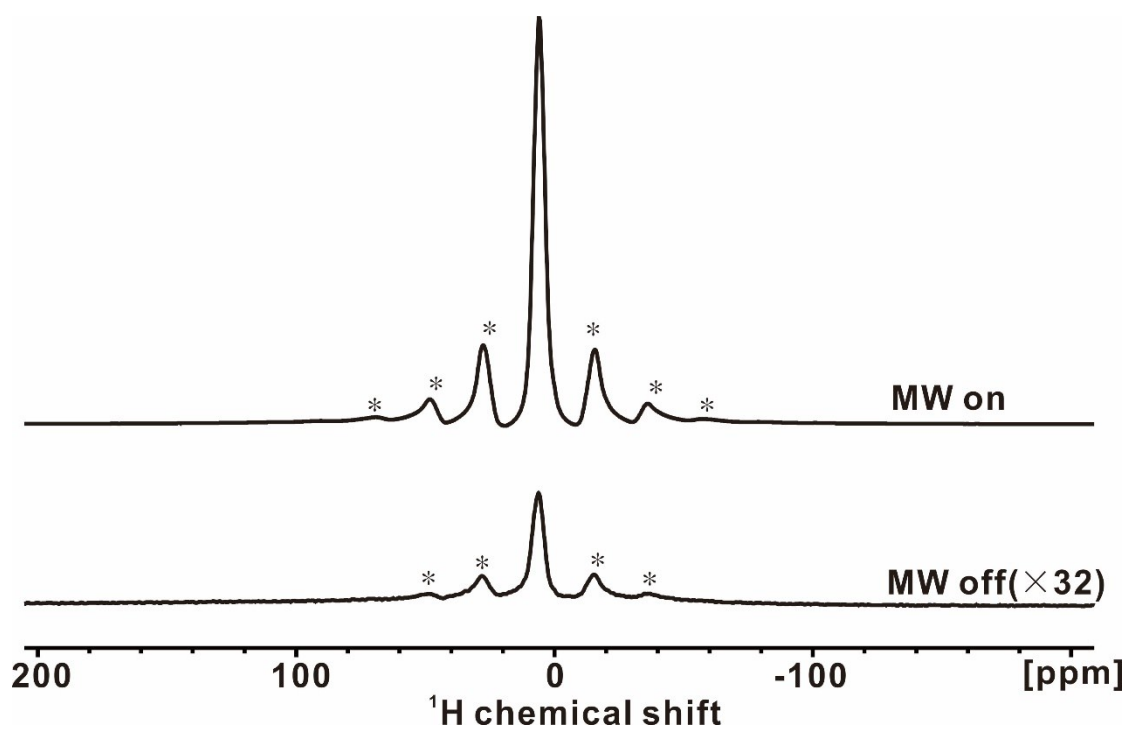
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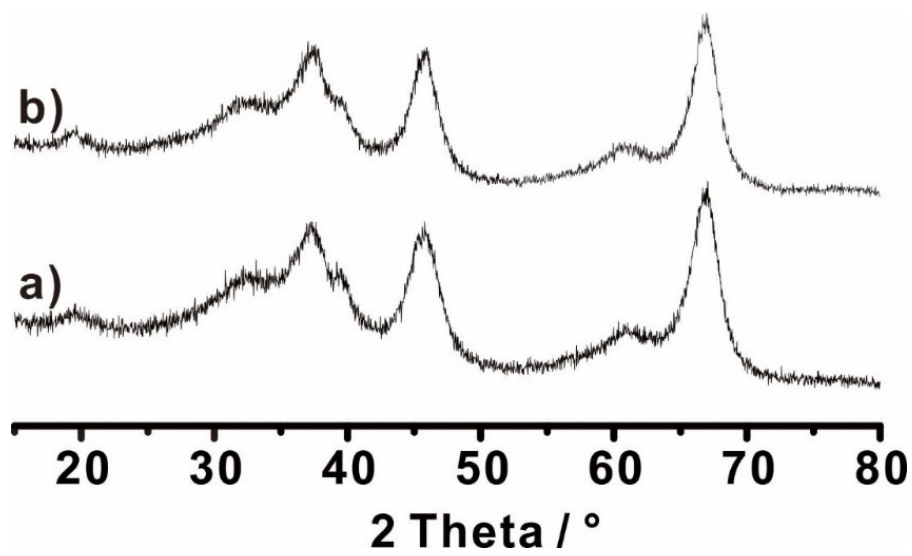
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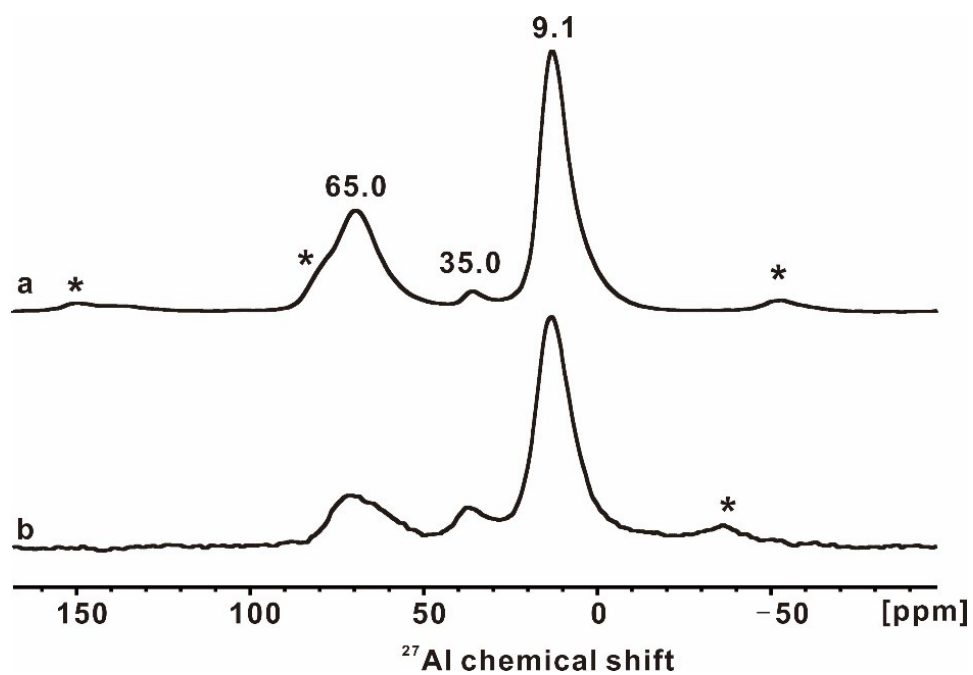
**Figure S1.**  $^1\text{H}$  MAS NMR spectra of dehydrated  $\gamma\text{-Al}_2\text{O}_3$  with microwave irradiation on (MW-on) or off (MW-off). A total of 4 scans were accumulated with a recycle delay of 10 s for each spectrum. The spinning speed was 8 kHz. \* denotes spinning sidebands.

**Table S1.** Isotropic chemical shift  $\delta_{CS}$  and second-order quadrupolar interaction parameter  $P_Q$  (where  $P_Q = C_Q(1 + \eta_Q^2/3)^{1/2}$  and  $C_Q$  and  $\eta_Q$  are the quadrupolar coupling constant and asymmetry, respectively) of each oxygen species on  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>(O) extracted from the 2D <sup>17</sup>O 3QMAS DNP spectrum.  $\delta_{F_2}$  and  $\delta_{F_1}$  represents the center of gravity of each specific species measured along direct dimension F<sub>2</sub> and isotropic dimension F<sub>1</sub>, respectively, on the sheared 2D spectrum in Fig. 3.

Site	$\delta_{F_2}$ /ppm	$\delta_{F_1}$ /ppm	$\delta_{CS}$ /ppm	$P_Q$ /MHz
Site 1	48.0	71.5	56.70	2.69
Site 2	68.4	69.6	68.80	0.61
Site 3	79.3	84.0	81.04	1.20



**Figure S2.** XRD patterns of parent  $\gamma\text{-Al}_2\text{O}_3$  (a) and  $\gamma\text{-Al}_2\text{O}_3(\text{O})$  (b).



**Figure S3.**  $^{27}\text{Al}$  MAS (a) and  $^1\text{H} \rightarrow ^{27}\text{Al}$  CP/MAS (b) NMR spectra of  $\gamma\text{-Al}_2\text{O}_3(\text{O})$ . \* denotes spinning sidebands. The spectra were recorded at 18.8T.